TRENDS IN MIREX RESIDUE LEVELS

IN LAKE ONTARIO SALMON - 1977 TO 1992

ALSO INCLUDED LEVELS FOR

PHOTOMIREX, DDT, DDD, DDE, PCB AND DIELDRIN

A Thesis

Presented to the Faculty of the Department of Biological

Sciences of the State University of New York

College at Brockport in Partial

Fulfillment for the degree

of Master of Science

by

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ABSTRACT

Chinook salmon (*Oncorhynchus tshawytscha*) collected from Lake Ontario during the fall of 1992 were analyzed for mirex, photomirex, DDT, DDD, DDE, PCBs and dieldrin. Mirex in fillet tissue ranged from 0.095 to 0.48 mg/kg (mean = 0.24 mg/kg). Analysis of variance (ANOVA) revealed no significant difference (P=0.285) between mean mirex residue values for fish collected in 1977, 1982, 1986 and 1992. However, analysis of covariance (ANCOVA), considering the covariate weight, indicated a statistically significant difference between 1977 and 1982, 1986 and 1992 mirex levels (P=0.001). Comparison of 1982, 1986 and 1992 by ANCOVA revealed no significant decrease in mirex levels. The following chlorinated hydrocarbons were also detected in fish tissue: photomirex (mean = 0.10 mg/kg), DDT (mean = 0.17 mg/kg), DDD (mean = 0.071 mg/kg), DDE (mean = 0.82 mg/kg) and PCB (mean = 0.85 mg/kg). Dieldrin was only detected in the egg samples.

ACKNOWLEDGMENTS

I would like to thank my major advisor, Dr. Joseph Makarewicz, for his assistance and guidance during this study. His patience with me was also very much appreciated. I would like to thank Dr. J. Haynes for his assistance in the sampling portion of this study. I would also like to thank Dr. J. Haynes and Dr. J. Hubbard for serving on my graduate committee.

I am very grateful to Mr. Ted Lewis for sharing his knowledge and experience with me during the course of my research. I am also very appreciative of his willingness to share his expertise in computer applications, because without his help this thesis would have been much longer in preparation.

A big thank you goes to Mr. Peter Papagellis for giving so much of his time, effort, knowledge and technical support to my study. Another big thank you goes to Mr. Joseph Guido for giving his time and sharing his expertise in statistical computer applications. I also would like to thank Mr. Andrew Boone for his support and for taking the time to review my thesis.

Last, but certainly not least, I wish to thank my parents. I am deeply grateful to them because without their support and encouragement this study, as well as my academic career at Brockport, truly would not have been possible.

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INTRODUCTION

The contamination of Lake Ontario salmon with mirex and other organochlorine compounds is well documented (Armstrong and Sloan 1980, Clark *et al.* 1984, Sloan 1987, Oliver and Niimi 1988, Niimi and Oliver 1989). Contamination of the lake with mirex and other organochlorine contaminants occurred primarily during the late 1960s and early 1970s. Although inputs into the lake ecosystem have declined (Task Force on Mirex [TFM] 1977, Warry and Chan 1981, Kuntz and Warry 1983, Durham and Oliver 1983, Halfron 1987), many Lake Ontario fish species still contain relatively high concentrations of these persistent contaminants (Whittle and Fitzsimons 1983, Borgmann and Whittle 1991, Suns *et al.* 1991).

Mirex, used as a pesticide and as a flame retardant, was produced by Hooker Chemicals and Plastics Corp. in Niagara Falls, N.Y., from 1959 to 1976 (Kaiser 1978). Elevated sediment concentrations of mirex along the south shore of Lake Ontario and into the eastern basin described by Holdrinet *et al.* (1978) indicate that both the Niagara and Oswego Rivers are the principal sources of mirex to Lake Ontario. Lake Ontario is the only Great Lake with significant mirex contamination (TFM 1977).

Mirex is a toxic, fully chlorinated organic hydrocarbon (Van Valin *et al.* 1968, Innes *et al.* 1969, Ludke *et al.* 1971, Bookhout *et al.* 1972, Ulland *et al.* 1977, Lue and de la Cruz 1978). Mirex is very resistant to biodegradation as it is not metabolized by most organisms (Ivie *et al.* 1974, Jones and Hodges 1974, Mehendale *et al.* 1972, Pritchard *et al.*1973, Pittman *et al.* 1976). Field and laboratory studies have demonstrated that mirex can be degraded photochemically in the environment and the primary photolytic derivative is 8-monohydro

mirex (photomirex) (Alley *et al.* 1974, Carlson *et al.* 1976, Mudambi and Hassett 1988). Photomirex, like mirex, is also resistant to biodegradation and is toxic (Villeneuve *et al.* 1979, Chu *et al.* 1981, Yarbrough *et al.* 1981).

Salmon are suitable as indicator organisms for assessment of lakewide contamination by organochlorine compounds because they are fast growing terminal predators, which exhibit migratory behavior and have a tendency toward a high degree of accumulation of organochlorine compounds (Norstom *et al.* 1978). Lake Ontario salmonids bioaccumulate mirex, PCBs and DDTs primarily through the food chain (Oliver and Niimi 1988, Borgmann and Whittle 1991), although less significant accumulation is possible for PCBs across the gills (Barber *et al.* 1991).

The accumulation of organic contaminants in fish tissue constitutes a serious ecological problem when considering these fish are eaten by piscivorous birds, mammals, and humans. In laboratory experiments by Hertzler (1988) and Daly *et al.* (1989), rats fed Lake Ontario salmon had significantly altered patterns of behavior versus rats fed salmon reared in the Pacific Ocean or rat chow. Epidemiological studies of mothers and their newborn infants exposed to significant quantities of lipophilic contaminants via consumption of fish from Lake Michigan, revealed that the concentrations of PCBs and other contaminants in their blood plasma were a direct function of the amount of fish consumed and the duration of exposure (Jacobson and Jacobson 1988). Infants born to mothers in high fish consumption groups had a decreased gestation period, were smaller and suffered from altered behavior (Fein *et al.* 1984, Jacobson *et al.* 1985). These effects persisted in some individuals for at least four years

(Jacobson *et al.* 1990). Fish eating birds have also experienced effects from contaminants, including embryologic mortality and deformities (Kurita *et al.* 1987).

While the worst contamination of Lake Ontario from the Niagara and Oswego Rivers occurred in the 1960s and early 1970s (Durham and Oliver 1983), there are still inputs of mirex and other organochlorine contaminants to the lake from both tributaries due to leaching from dump sites (Warry and Chan 1981, Scrudato and Delprete 1982, Whittle and Fitzsimons 1983, Kuntz and Warry 1983, Kauss 1983, Halfron 1987). In addition, Lewis and Makarewicz (1988) demonstrate that salmon have contaminated Lake Ontario tributaries via their spawning migrations, both through direct release as well as through ingestion of salmon tissue and subsequent transport by stream organisms. The recycling of mirex back into Lake Ontario from these tributaries contaminated from spawning is estimated to be potentially 26 gm/yr (Lewis and Makarewicz 1988).

The main objectives of this study were to determine current mirex residue levels in Lake Ontario chinook salmon; to compare existing levels with past data to determine the trend in mirex residue levels over a 15 year period; and to analyze the fish tissue for additional chlorinated hydrocarbons (DDT, DDD, DDE, dieldrin and PCB) to establish baseline level in chinook salmon.

METHODS

Chinook salmon (*Oncorhynchus tshawytscha*) were collected by electroshocking from Sandy Creek and Oak Orchard Creek, both tributaries of Lake Ontario. A total of 12 fish were sampled during the fall spawning run of 1992. Four fish from each of three different weight intervals, <1.8, 1.8-3.6, and >3.6 kg (corresponding to intervals of <4, 4-8, and >8 lb., respectively), were analyzed. The length (mm), weight (kg) and sex were noted, and scales were collected for each fish sampled. Fish were then packed in ice and transported to the laboratory. A standard fillet from each fish (i.e., entire side of fish from just behind the operculum to tail, including skin, bones of half the rib cage and one pelvic fin, but excluding the vertebral column, dorsal, pectoral, anal and caudal fins) (Armstrong and Sloan 1980) was wrapped in aluminum foil and frozen. Eggs samples were also frozen.

After thawing, a 5 gram aliquot of each egg and tissue sample was mixed well with 20 grams of anhydrous sodium sulfate. The sample was extracted overnight with 75 ml of methylene chloride/hexane (20:80 v/v) by Soxhletic extraction before lipids were removed in a Florisil column. Two different solvent mixtures, methylene chloride/hexane (20:80 v/v) and methylene chloride/hexane/acetonitrile (50:49.6:0.35 v/v/v), were eluted through the florisil column, 40 ml at a time. After each solvent elution, the sample was concentrated, but never allowed to go to dryness. Prior to analysis, samples were brought to volume (1 ml) in hexane. Sample lipid content was determined by Soxhlet extraction followed by evaporation to a constant weight. All reagents used were pesticide grade.

Mirex, photomirex, DDT, DDD, DDE, dieldrin and PCB Aroclors 1254/1260 were analyzed on a Hewlett Packard Gas Chromatograph (Model 5890) equipped with a Ni^{63} electron capture detector, an autosampler (Model 7673A), and a HP3396A integrator. Injections (1 µl) were split 50:1 with the injection port temperature at 222° C. The initial oven temperature was 80° C and increased 5° C/min until a final temperature of 275° C was reached and held for 40 minutes. The detector temperature was 300° C. The chromatographic column was a Supelco PTE-5 fused silica capillary column (30m x 0.25mm x 25µm film coating). The carrier gas was argon/methane (95:5%). A combination standard of 0.1 mg/kg mirex and photomirex, and 0.5 mg/kg DDT, DDE, DDD and dieldrin were analyzed with each set of samples.

GC/MS Confirmation

Confirmation of compounds was achieved with a Hewlett Packard 5890 series II gas chromatogragh, equipped with a Hewlett Packard 5970B mass selective detector (GC/MS) and a J&W DB-5 wide bore column (15 meter with a 0.25 µm film coating). Injections (2 µl) were splitless for the first 30 s, with the injection port at 350° C. The initial oven temperature was 100° C and increased 15° C/min until a final temperature of 350° C. The mass selective detector was tuned with an EM voltage of 2000 v, while samples were run at 2200 to increase sensitivity. Samples were analyzed using selective ion monitoring with a dwell time of 20 milliseconds. The ion range scanned for mirex and photomirex was 270-276, including ions 270, 272, 274, and 276. The ion scan range for DDE was 246-250; for DDT and DDD, it was 232.7-237.7; and for dieldrin, it was 378-382.

Representative standard and sample chromatograms for the GC with an electron capture detector (GC/EC) are shown in Figures 1 and 2, respectively. A representative GC/MS chromatogram and mass spectra, using selective ion monitoring, for a mirex standard and mirex in a sample, are shown in Figures 3 and 4, respectively.

Quality Control

Quality control procedures included analysis of reagent blanks, replicate sample analysis for comparison of techniques (Appendix I) and replicate analysis for estimating reproducibility of results (Appendix II). Sample spike recovery efficiencies were also determined (Appendix III).

RESULTS

Since this study encompassed 15 years of analysis, often by different methods (packed vs. capillary GC), fish tissue analyzed and stored by Insalaco *et al.* (1982) was analyzed using methodology described here. No significant difference ($P>0.05$) was observed for the two separate analyses by two different techniques (Appendix I). A comparison of the average concentrations and ranges observed in fish tissue samples for both the GC/EC and GC/MS indicate excellent agreement (Appendix IV). Recovery efficiencies for DDT, DDD, DDE and dieldrin were 105.6, 94.6, 121.3 and 118.8 %, respectively (Appendix III). Recovery efficiencies for mirex and photomirex were 95 %. Replicate analyses (n=5) indicated precision was reasonable (% RSD=20 to 37) (Appendix II). Even though a sample was homogenized, it was still somewhat heterogeneous and this may be the cause of the variability.

Fish age, weight and length ranges were as follows: 1 to 3 years old; 1.9 to 12.2 kg (mean = 7.12 kg); and 560 to 1040 mm (mean = 826 mm) (Table 1, Appendix V). Mirex, photomirex, DDT, DDD, DDE, PCBs were observed in all fish tissue and egg samples (Table 2). Dieldrin was not detected in any of the fish samples analyzed on the GC/EC or the GC/MS, but was observed in the eggs (Table 2, Appendix V). Lipids content ranged from 1.92 to 7.85 percent lipid (mean = 3.9%) (Table 1).

Much of the variability in concentration in the dependent variables mirex ($r^2=0.59$), photomirex ($r^2=0.50$), DDT ($r^2=0.51$), DDD ($r^2=0.41$), DDE ($r^2=0.51$), total DDT ($r^2=0.50$) and PCB ($r^2=0.52$) was explained by the independent variable fish weight (Table 3, Figures 5 and 6). A relationship between length and contaminant concentration also existed, but was not

as strong as weight (Table 4). The lack of a significant relationship between lipid content and contaminant concentration observed here (Table 5) has been observed previously in spawning salmon (Waldina *et al.* 1973, DeVault and Weishaar 1982, Murray 1991).

DISCUSSION

Mirex Trend Analysis

Analysis of variance (ANOVA) revealed no significant difference (P=0.285) between mean mirex residue values for fish collected in 1977, 1982, 1986 and 1992 (Table 6). However, interannual comparisons of mean mirex concentrations do not consider the significant effect of weight on mirex concentration (Table 7). Plotting the fish weight versus mirex concentration for each of the four studies (Figure 7) reveals this linear relationship and indicates a decrease in the elevation of each line from 1977 to 1992 (Figure 8).

To determine if mirex levels were significantly different between years, an analysis of covariance (ANCOVA) was undertaken with weight being the covariate. Using SAS (Statistical Analysis System, SAS Institute Inc.), an ANCOVA model was created and designed to evaluate mirex concentration across all weights for all years. The interaction between weight and group in the ANCOVA model was not significant indicating that the slopes of all four regression lines were not significantly different (P=0.978) (Table 8). The model then determined whether there was any significant difference in mirex concentration data between groups (years) across all weights. This is essentially comparing the vertical distance between lines across all weights. There was a significant difference found in mirex concentration between groups (years) (P=0.001) (Table 9). Using the Scheffe Test (Zar 1984), a significant difference in mirex concentration was found to occur only when the data from 1977 was contrasted to each other year. No significant difference occurred when any of the other years (1982, 1986 and 1992) were contrasted to each other. The ANCOVA model

generated estimated Y-intercepts and one common slope, which were used to create a weight adjusted equation for each of the years. The weight adjusted regression lines clearly show the significant decrease in elevation from 1977 to 1982. Following 1982, the decreases in elavation are not significant (Figure 9).

These results concur with those of the New York State DEC's contaminant trend analysis of Lake Ontario Salmon tissue, which suggests mirex levels may have peaked in the late 1970s with a decreasing trend starting in subsequent years (Armstrong and Sloan 1980). This decrease in mirex concentration in the late 1970s could be reflective of the ban on mirex production in 1976. Sloan (1987) reports mirex levels in Lake Ontario fish tissue from 1981 to 1986 remained relatively stable, which supports my conclusion of no significant decline in mirex after 1982. A decline in the rate of decrease in mirex concentration levels starting in the early 1980s is evident (Figure 10).

Lake Ontario Photomirex / Mirex Ratios

The photomirex to mirex ratio is a measure of the mirex availability in the euphotic zone (Great Lakes Water Quality Board 1987b). Mudambi *et al.* (1992) provide strong evidence that photomirex present in the Lake Ontario ecosystem is only formed by the photolysis of mirex present in the lake surface waters by sunlight with possibly a small contribution from photolysis in the river waters. This process is accelerated in the presence of humic acids (Mudambi and Hasset 1988). Mirex concentration levels in Lake Ontario biota have decreased since the late 1970s (Norstom *et al.* 1980a, Armstrong and Sloan 1980, Murray 1991), and assuming photolytic degradation of mirex has occurred, it is possible that the photomirex to mirex (p/m) ratios could have increased since this time period. However, Norstrom *et al.*

(1980a) analyzed herring gull eggs taken from Lake Ontario colonies and found that the p/m ratio (0.35-0.42) remained relatively constant from 1972-1978, although the mirex levels in Lake Ontario biota declined. They concluded that once mirex and photomirex enter the food chain in the lake ecosystem, these compounds are protected from further photodegradation by being sequestered and cycled within and between the various food webs.

The p/m ratios show little fluctuation within species (Kaiser 1978), and remain relatively constant among different species in the higher levels of the food chain for a given system (Oliver and Niimi 1988). Several p/m ratios for fish species sampled from Lake Ontario are given in Table 10. Interpretation of the data listed in Table 10 with respect to trends in p/m ratios is difficult. The p/m ratios appear to have remained relatively stable, with a range of approximately 0.4 to 0.6 currently observed in Lake Ontario fish.

Trends in other Contaminants

PCBs

PCBs were commercially manufactured in the United States starting in 1929 and used widely in industry primarily as heat transfer fluids in transformers and capacitors (NYDEC 1985). The most commonly used PCB mixtures in the United States were those marketed by Monsanto under the trade name Aroclor. PCBs are toxic compounds which have low aqueous solubility, are resistant to degradation in the environment, and bioaccumulate in both terrestrial and aquatic food chains (Council of Great Lakes Research Managers 1985, Taylor *et al.* 1989, Borlakoglu and Haegele 1991, Seegal et al. 1991). Restrictions on use of PCBs started in the early 1970s, followed by cessation of production in 1977 (Armstrong and Sloan 1980). Following the ban on PCBs, a subsequent decline in the concentration of PCBs in Lake

Ontario fish tissue took place in the late 1970s (Armstong and Sloan 1980, Great Lakes Water Quality Board 1987a, Suns *et al.* 1991). Armstrong and Sloan (1980) report mean total PCB concentrations in chinook salmon from Lake Ontario for 1977 and 1978 of 8.48 and 4.80 mg/kg, respectively. The decline of the late 1970s leveled off in the 1980s, and subsequent data was difficult to interpret with respect to consistent trends (Sloan 1987, Great Lakes Water Quality Board 1989, Borgmann and Whittle 1991). Skinner (1991) indicates that since the decrease in PCB levels between 1979 and 1980, coho salmon demonstrate a static pattern with no change in PCB levels evident through 1990.

Oliver and Niimi (1988) found the chlorine content of the PCBs increases at higher trophic levels in the Lake Ontario food chain. Over 50 % of the total PCBs in Lake Ontario fish tissue is equivalent to Aroclor 1254, which contains predominantly tetra-, penta- and hexachlorinated isomers. The second most prevalent is Aroclor 1260 (Armstrong and Sloan 1980, Newell *et al.* 1987). In the present study, I analyzed for total PCBs equivalent to Aroclors 1254 and 1260. Recent analysis of chinook salmon tissue from Lake Ontario by the NYDEC indicates there is still significant Aroclor 1254/1260 residue levels, ranging from 0.19 to 4.37 mg/kg, and less chlorinated Aroclors 1016 and 1248 are present at very low levels (Table 11). My PCB residue levels ranged from 0.32 to 1.7 mg/kg and concur with NYDEC's results (Table 11). There appears to be no current downward trend in the levels of Aroclors 1254/1260 in Lake Ontario salmon.

DDT

DDT, introduced during World War II for the control of insect-transmitted diseases, was used widely as an insecticide for agricultural and forest pest control. Because of its toxicity

and ability to accumulate in nontarget organisms, the use of DDT was banned in New York State in 1971 and the United States in 1973 (Johnson 1968, NYDEC 1985, Newell *et al.* 1987, Rehana and Rao 1992). Similar to mirex and PCBs, DDT is relatively stable and persistent in the environment (Armsrong and Sloan 1980). However, in the environment and in animals, DDT is degraded primarily to DDE and DDD (Environment Canada 1991). In response to the ban on DDT, residues in Lake Ontario fish declined markedly during the 1970s (Armstrong and Sloan 1980, Borgmann and Whittle 1991, Suns *et al.*1991). Armstrong and Sloan (1980) report mean total DDT concentrations in smallmouth bass from Lake Ontario for 1969 to 1970 of 6.09 mg/kg and for 1975 to 1980 of 0.33 mg/kg. Similar to mirex and PCB levels, the rate of decline in DDT residue levels in Lake Ontario fish decreased in the 1980s compared to the 1970s (Borgmann and Whittle 1991, Great Lakes Water Quality Board 1989).

The most prevalent DDT metabolite in the Great Lakes ecosystem is p,p-DDE (Environment Canada 1991). DDE is the primary aerobic degradation product of DDT and is extremely stable, while DDD, through a series of aerobic and anaerobic reactions, may be further degraded (Devault *et al.* 1988). DDE usually represents approximately 80% of the total DDT measured (Great Lakes Water Quality Board 1987a). DeVault *et al.* (1988) reported DDE levels which were 80% to 90% of the total DDT in coho salmon sampled from Lake Ontario in 1984. Suns *et al.* (1991) reported DDE values which were generally 81% to 100% of the total DDT residues for spottail shiners collected from Lake Ontario from 1975 to 1987. DDE levels from the present study represent on average 77% of the total DDT residues. DeVault *et al.* (1988) report DDD contributing 1% to 8% of the total DDT for Lake

Ontario coho. DDD from the present study represents on average 7% of the total DDT. The presence of DDD could indicate a more recent source of DDT, since it is less stable than DDE (Aguilar 1984).

Recent analysis of chinook salmon tissue from Lake Ontario by the NYDEC indicate that DDT, DDD, and DDE are still detected with levels ranging from <0.002 to 0.91 mg/kg (Table 12). Results from the present study for residue levels of DDT, DDD, and DDE ranged from 0.079 to 1.7 mg/kg and concur with the NYDEC's results (Table 12). There appears to be no current downward trend in the levels of DDTs in Lake Ontario salmon.

Recent analysis of salmon sampled in the fall 1991 from Lake Ontario by the NYDEC (Skinner 1992) indicate the concentration of mirex in salmon tissue continues to exceed the U.S. Food and Drug Administration (FDA) action level of 0.1 mg/kg for human consumption. The results of my study also show that levels are still well in excess of this guideline. The PCB and total DDT residue levels in Lake Ontario salmon from my study are, on the average, below the FDA action levels, which are 2.0 and 5.0 mg/kg, respectively. However, my results reported for both PCB and total DDT exceed guidelines published by Newell *et al.* (1987) for protecting fish-consuming wildlife, which are 0.11 mg/kg for PCB and 0.2 mg/kg for DDT residues.

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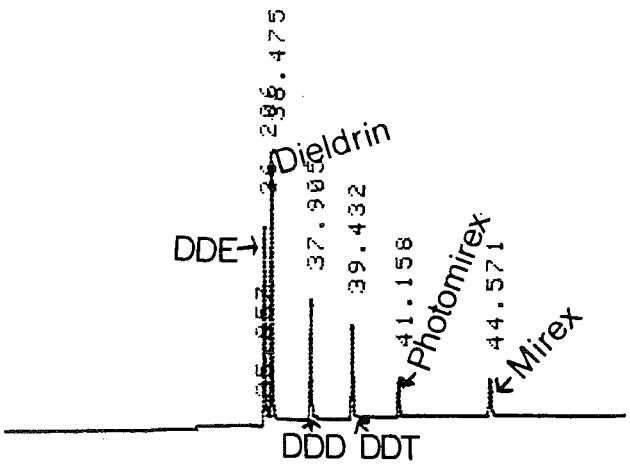

Figure 1. Representative chromatogram from the GC/EC of standard mixture. Chromatographic conditions are given in text.

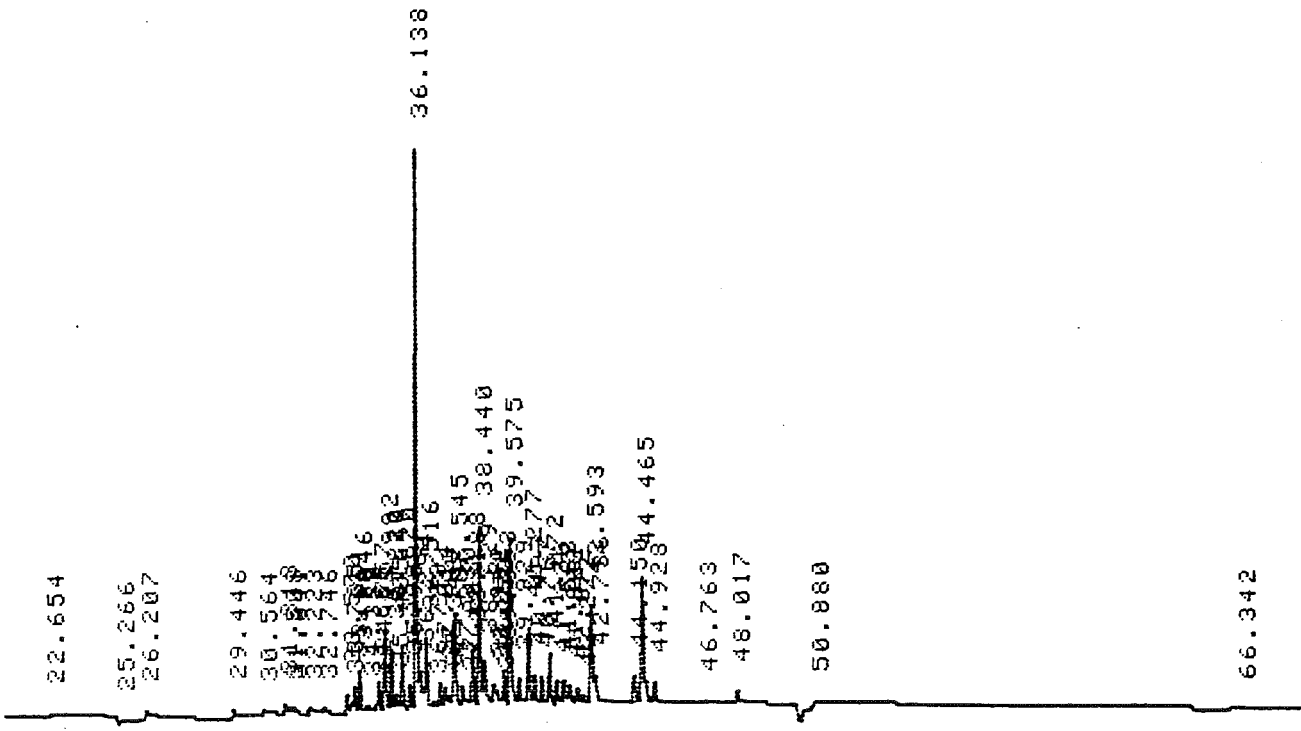

Figure 2. Representative chromatogram from the GC/EC of sample extract. Chromatographic conditions are given in text.

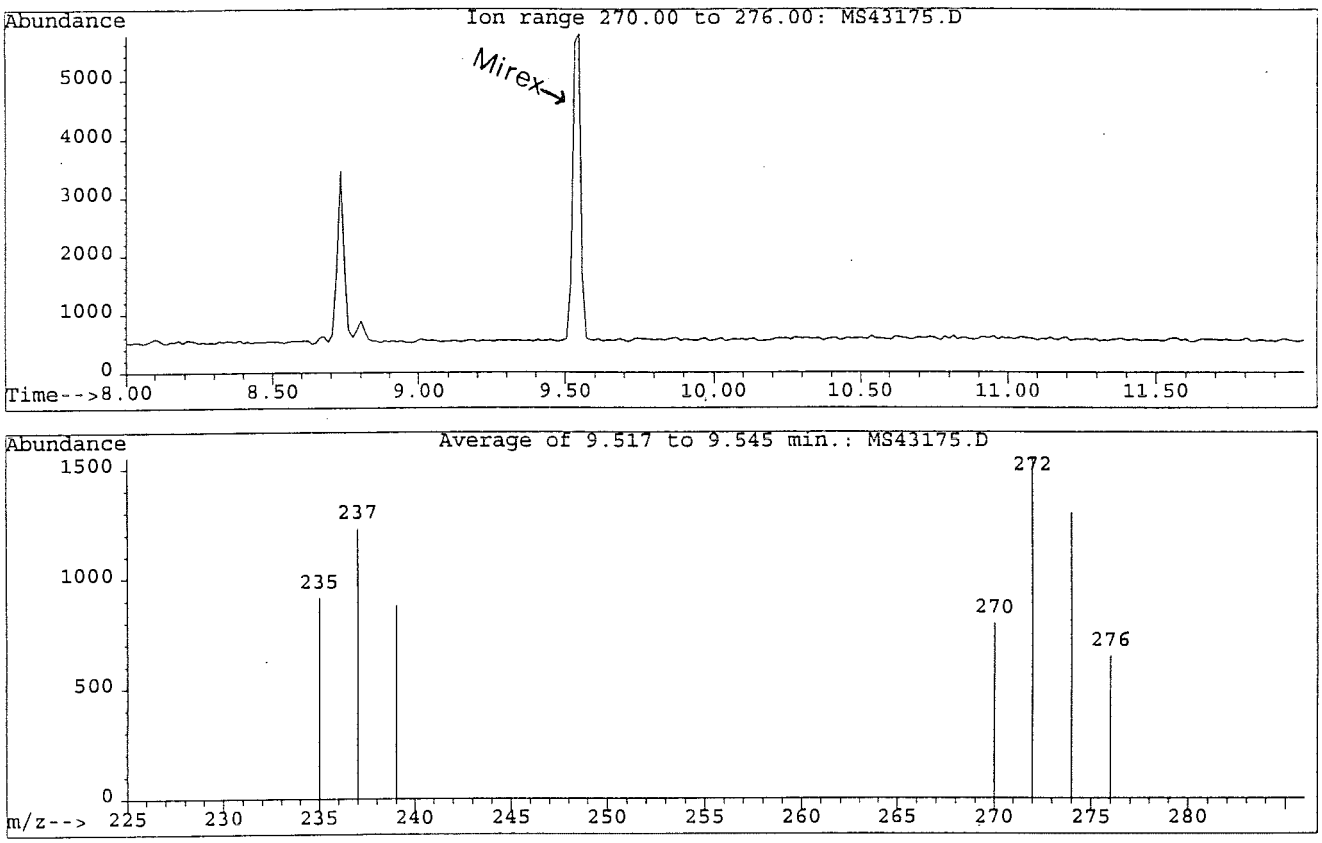

Figure 3. Representative chromatogram and mass spectra from the GC/MS of a mirex standard. Chromatographic conditions are given in text.

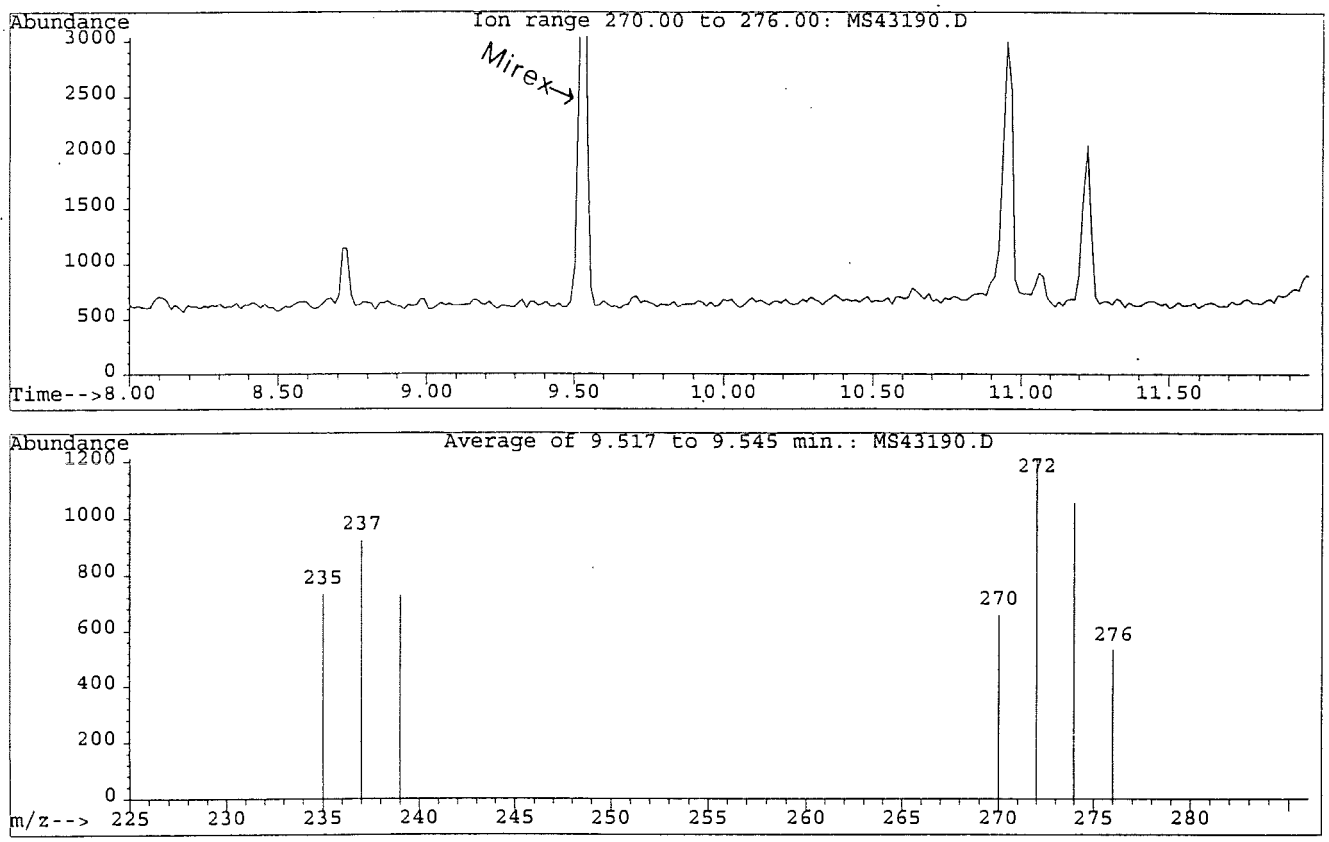

Figure 4. Representative chromatogram and mass specra from the GC/MS of mirex in a sample. Chromatographic conditions are given in text.

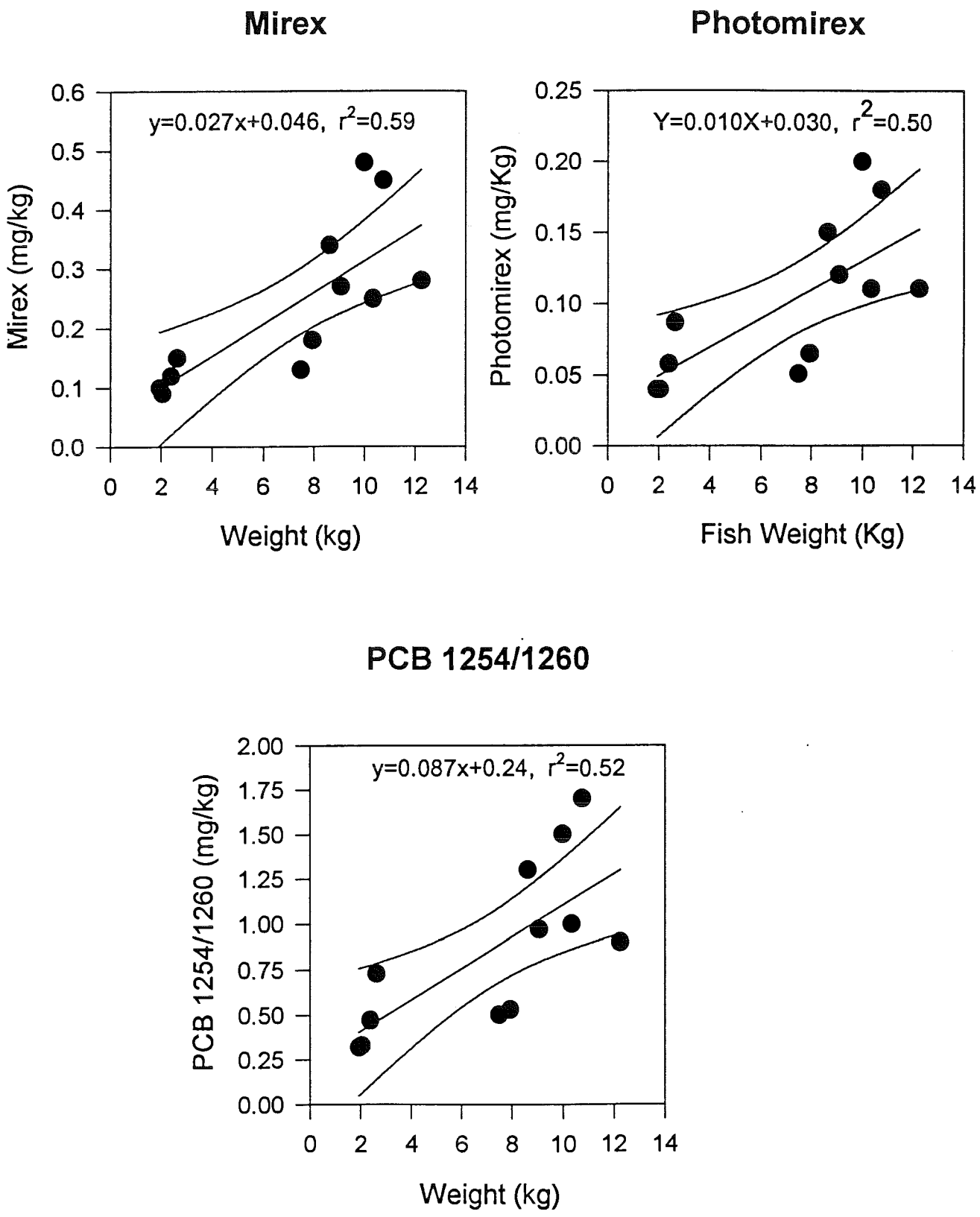

Figure 5. Regressions of the contaminants mirex, photomirex and PCBs (dependent variables), versus fish weight (independent variable). Data from the 1992 fish collection. Error bars are the 95% confidence intervals.

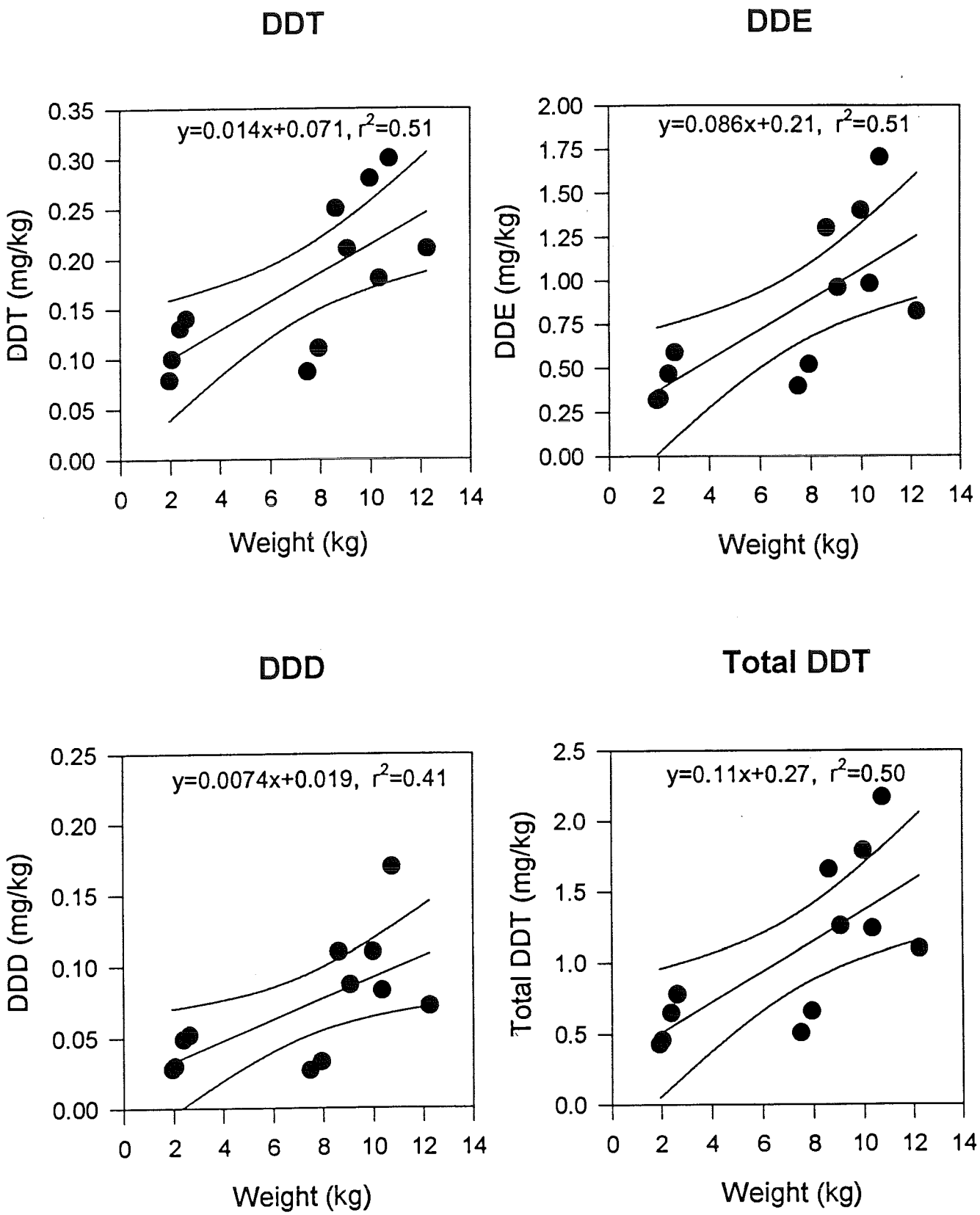

Figure 6. Regressions of the contaminants DDT, DDD, DDE and Total DDT (dependent variables), versus fish weight (independent variable). Data from the 1992 fish collection. Error bars are the 95% confidence intervals.

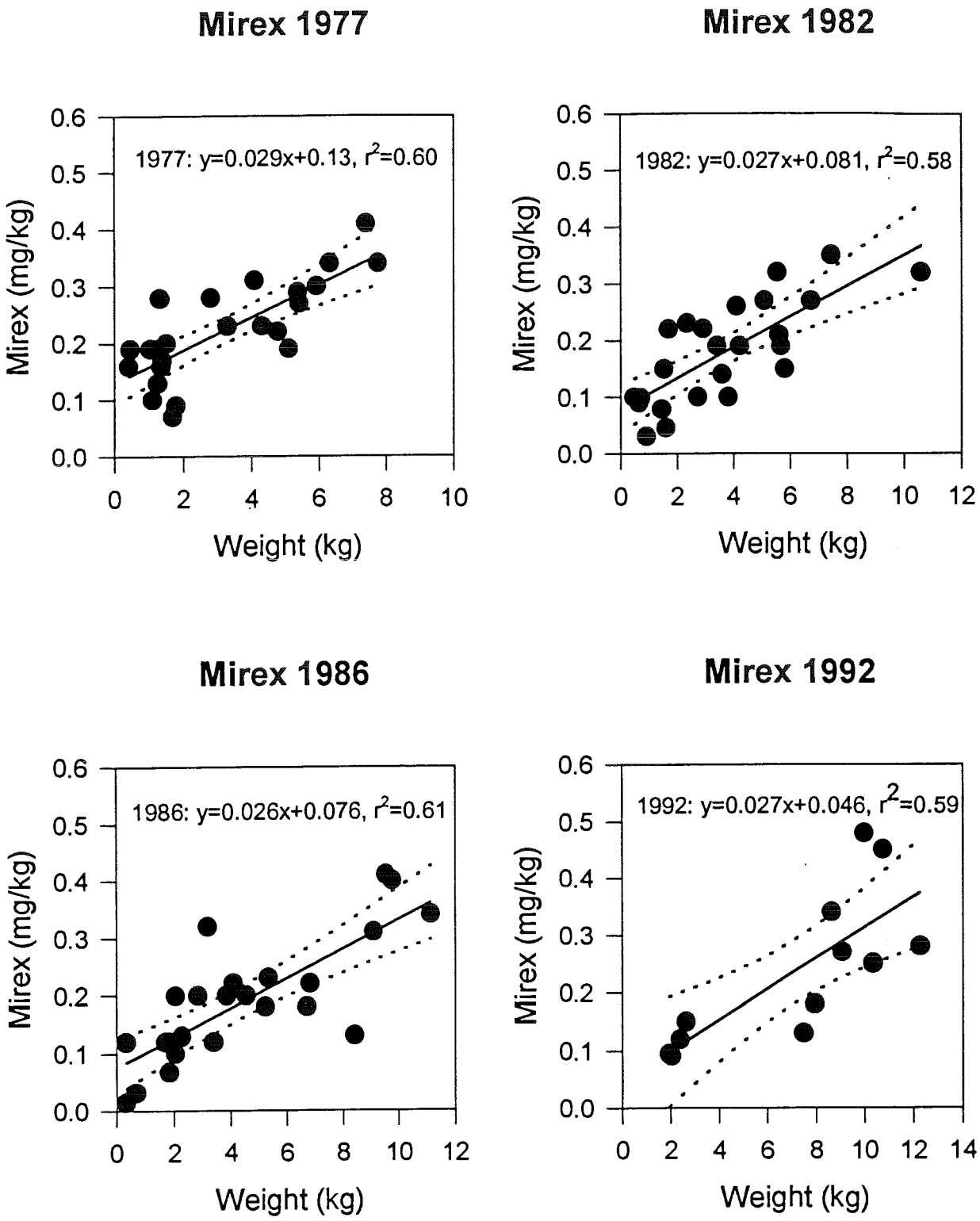

Figure 7. Regressions of mirex concentration versus fish weight for 1977, 1982, 1986 and 1992 fish collections. Error bars are the 95% confidence intervals.

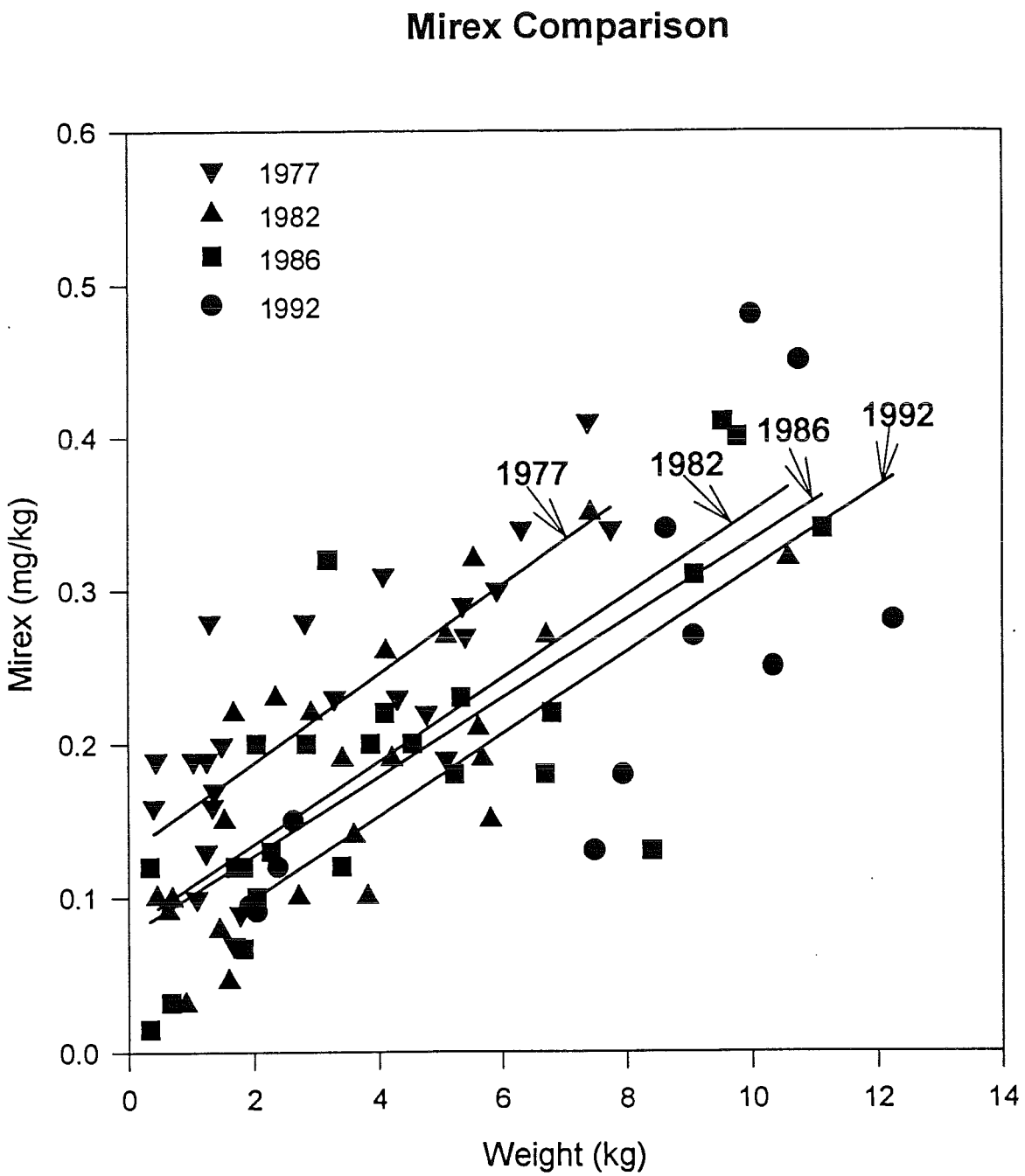

Figure 8. Comparison of regression lines of mirex concentration versus fish weight for 1977, 1982, 1986 and 1992 fish collections.

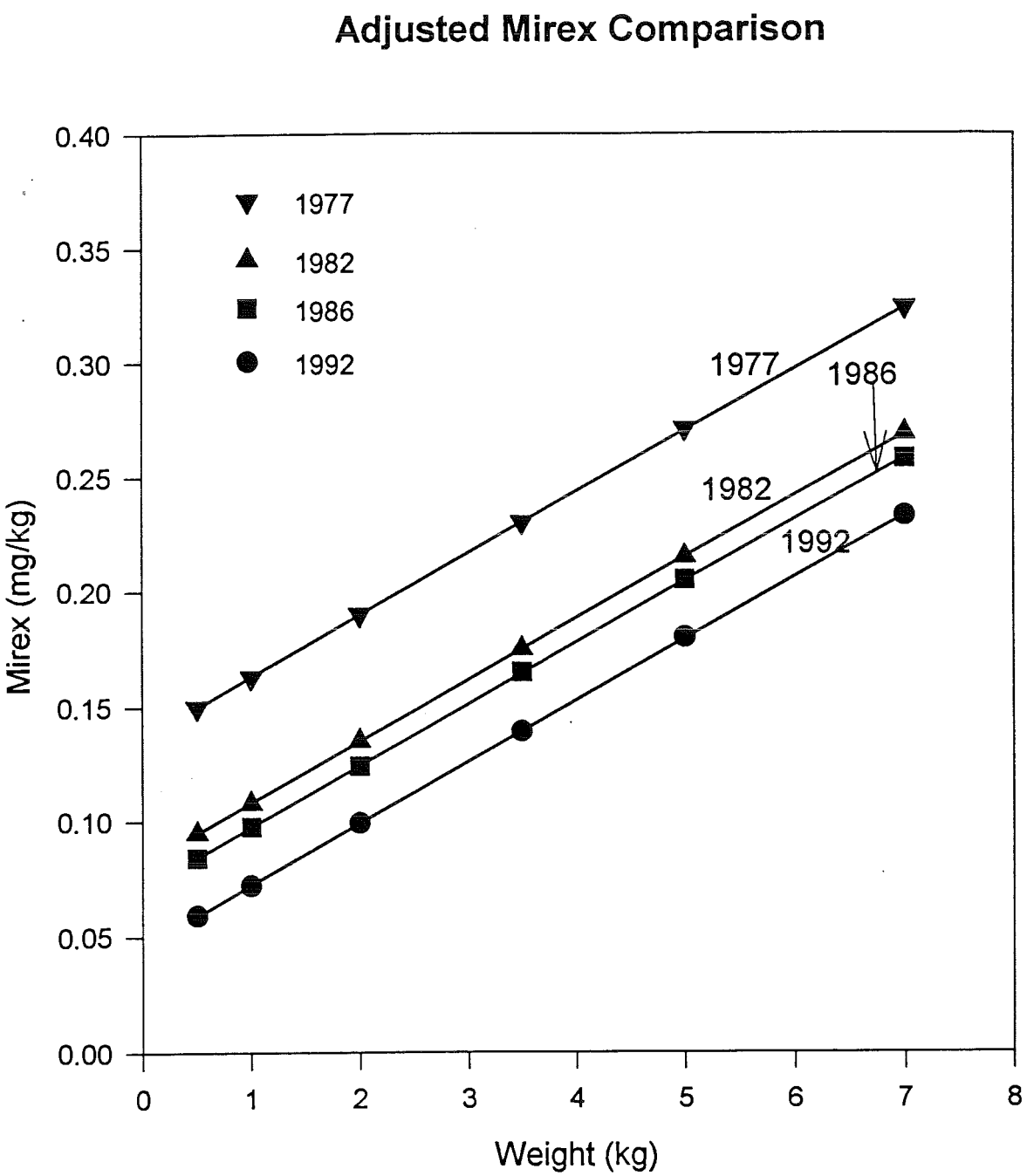

Figure 9. Comparison of regression lines of weight adjusted mirex concentrations versus fish weight for 1977, 1982, 1986 and 1992 fish collections.

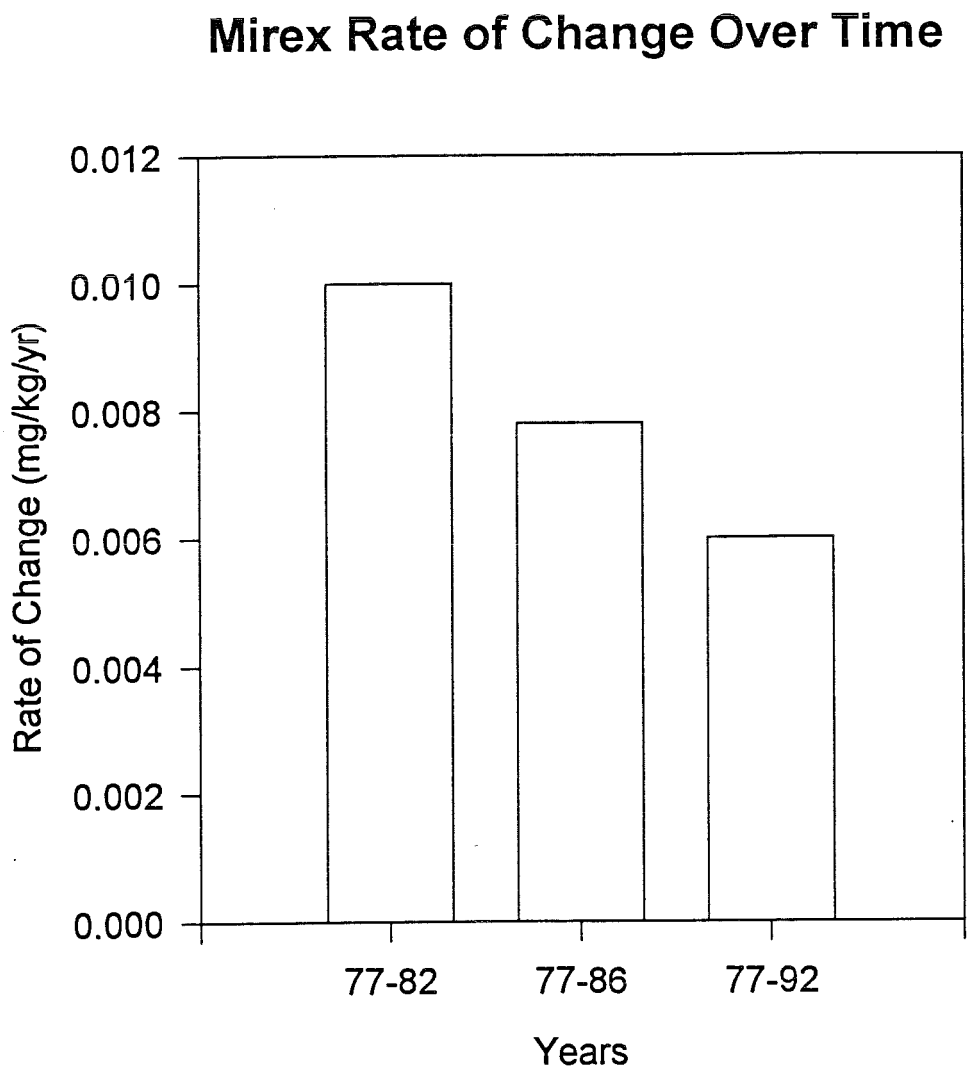

Figure 10. Comparison of the rate of change in mirex levels versus time in fish collected in 1977, 1982, 1986 and 1992.

Table 1. Average fish weight, length and lipid content. Numbers in parentheses are standard errors.

	% Lipids	Weight	Length
Fish (n=12)	3.9 (0.55)	7.1 (1.10)	826 (53.2)
Range	1.9-7.9	1.9-12.3	560-1040
Eggs (n = 2)	11.7	--	--
Range	10.7-12.7	--	--

Table 2. Mirex, photomirex, DDT, DDD, DDE, total DDT, PCB and dieldrin concentration in fish and egg tissue. Numbers in parentheses are standard errors. All results are in mg/kg.

	Mirex	Photo-mirex	DDT	DDD	DDE	Total DDT	PCB 1254/1260	Dieldrin
Fish (n=12)	0.24 (0.038)	0.10 (0.016)	0.17 (0.022)	0.071 (0.013)	0.82 (0.29)	1.06 (0.17)	0.85 (0.13)	ND
Range	0.095-0.48	0.04-0.20	0.079-0.3	0.027-0.17	0.32-1.7	0.43-2.2	0.32-1.7	--
Eggs (n=2)	0.19	0.10	0.20	0.092	1.20	1.50	1.09	0.061
Range	0.16-0.22	0.091-0.11	0.18-0.21	0.085-0.099	1.0-1.4	1.3-1.7	0.098-1.2	0.059-0.06

ND - Not Detected

Table 3. Linear regression analysis of contaminant concentration (dependent variable) on fish weight (independent variable), n = 12. All equations are significant at $P<0.01$.

	Coefficient of Determination (r^2)	Predictive Equation
Mirex	0.59	0.027(weight) + 0.046
Photomirex	0.50	0.01(weight) + 0.030
DDT	0.51	0.014(weight) + 0.071
DDD	0.41	0.0074(weight) + 0.019
DDE	0.51	0.086(weight) + 0.21
Total DDT	0.50	0.11(weight) + 0.27
PCB 1254/1260	0.52	0.087(weight) + 0.24

Table 4. Linear regression analysis of contaminant concentration (dependent variable) on fish length (independent variable), n = 12. All regressions are significant at $P<0.05$

	Mirex	Photomirex	DDT	DDD	DDE	PCB
Coefficient of Determination (r^2)	0.51	0.44	0.46	0.35	0.47	0.45

Table 5. Linear regression analysis of contaminant concentration (dependent variable) on percent lipids (independent variable), n = 12. There are no significant regressions ($P>0.05$).

	Mirex	Photomirex	DDT	DDD	DDE	PCB
Coefficient of Determination (r^2)	0.16	0.15	0.19	0.14	0.11	0.14

Table 6. Mean mirex concentrations in fish tissue collected in 1977, 1982, 1986 and 1992, and the results of ANOVA.

	1977 n=24	1982 n=24	1986 n=24	1992 n=12	F-Value	Level of Significance
Mean mirex (mg/kg)	0.22 (0.018)	0.18 (0.018)	0.19 (0.021)	0.24 (0.038)	1.3	0.29
Range	0.070-0.41	0.031-0.35	0.015-0.41	0.091-0.48		

Table 7. Mean fish weights in 1977, 1982, 1986 and 1992, and the results of ANOVA. The Tukey Test indicates significant differences occurred between 1992 and 1977, and between 1992 and 1982 mean fish weights. Numbers in parentheses are standard errors.

	1977 n=24	1982 n=24	1986 n=24	1992 n=12	F-Value	Level of Significance
Mean weight (kg)	3.2 (0.47)	3.7 (0.52)	4.4 (0.66)	7.1 (1.1)	5.3	0.0021
Range (kg)	0.41-7.8	0.46-10.6	0.34-11.1	1.9-12.2		

Table 8. Slopes of regression lines for respective years of the relationship between fish weight and mirex concentration (ANCOVA). Results from the ANCOVA model for the interaction between weight and group (year) determined there was no significant difference between slopes.

	1977 n=24	1982 n=24	1986 n=24	1992 n=12	F-Value	Level of Significance
Slope	0.029	0.027	0.026	0.027	0.070	0.98

Table 9. Mean weight adjusted mirex concentrations for fish collected in 1977, 1982, 1986 and 1992 from the ANCOVA model. Results from the ANCOVA model show a significant difference in mirex concentration data between groups (years) across all weights. Using the Scheffe Test, a significant difference was found to occur between 1977 and all other years. No significant difference occured between 1982, 1986 and 1992.

	1977 n=24	1982 n=24	1986 n=24	1992 n=12	F-Value	Level of Significance
Adjusted mean (mg/kg) mirex	0.25	0.20	0.18	0.16	6.3	0.0010

Table 10. Photomirex to mirex (p/m) ratios for fish species sampled from Lake Ontario.

	Fish Species collected in 1977 (Norstrom *et al* 1980b)	Fish Species collected in 1982 (McDowell *et al.* 1986)	Fish Species collected in 1983 and 1984 (Great Lakes Water Quality Board 1987b)	Salmon collected in 1986 (Murray 1991)	Salmon collected in 1992 (present study)
P/M Ratio	0.3 to 0.4	0.60	0.38 and 0.43	0.42	0.43

Table 11. Average concentration of PCBs (Aroclor 1254/1260) in chinook salmon tissue. Data are from the analysis of two fish collections by the NYSDEC in 1989 and 1991 and from fish collected for the present study in 1992. Numbers in parentheses are the ranges.

PCB Aroclor	Fall Chinook 1989 (Skinner 1990) n=29	Fall Chinook 1991 (Skinner 1992) n=30	Fall Chinook 1992 present study n=12
Weight (kg)	9.9 (6.7-11.5)	9.7 (7.1-11.3)	7.1 (1.9-12.3)
1254/1260 (mg/kg)	1.14 (0.19-4.37)	1.64 (0.83-2.62)	0.85 (0.32-1.7)
1016/1248 (mg/kg)	0.030 (<0.020-0.120)	0.052 (<0.020-0.150)	not determined

Table 12. Average concentration of DDT, DDD and DDE in chinook salmon tissue. Data are from the analysis of two fish collections by the NYSDEC in 1989 and 1991 and from fish collected for the present study in 1992. Numbers in parentheses are the ranges.

	Fall Chinook 1989 (Skinner 1990) n=29	Fall Chinook 1991 (Skinner 1992) n=30	Fall Chinook 1992 present study n=12
Weight (kg)	9.9 (6.7-11.5)	9.7 (7.1-11.3)	7.1 (1.9-12.3)
DDT (mg/kg)	0.054 (<0.002-0.18)	0.061 (0.024-0.093)	0.17 (0.079-0.30)
DDD (mg/kg)	0.025 (<0.002-0.079)	0.036 (0.006-0.056)	0.071 (0.027-0.17)
DDE (mg/kg)	0.54 (0.14-0.91)	0.51 (0.26-0.83)	0.82 (0.32-1.7)

Appendix I

Evaluation of 1993 Analytical Techniques

Replicate mirex analyses (1993, n = 11) were performed on a previously analyzed chinook salmon tissue (T3A, Insalaco 1977).

	1993	1977
Mean Mirex (mg/kg)	0.40	0.41
Std Dev	0.10	

Results of the comparison of 1993 and 1977 mean mirex concentrations using the T-Test.

Calculated T value	Critical T value	Level of Significance
0.35	2.2	P > 0.05

Appendix II

Results of replicate analyses of fish sample #1

Replicate analyses were performed on fish sample #1 for mirex, photomirex, DDT, DDD, DDE and PCB. The replicates were given the following sample ID's; 1A, 1B, 1C, 1D and 1F. All results are in mg/kg. (%RSD = percent relative standard deviation)

	Mirex	Photomirex	DDT	DDD	DDE	Total DDT	PCB 1254/1260
1A	0.23	0.098	0.18	0.082	0.77	1.0	0.85
1B	0.24	0.10	0.17	0.064	0.82	1.1	0.84
1C	0.27	0.12	0.20	0.096	0.93	1.2	1.0
1D	0.24	0.10	0.17	0.060	0.79	1.0	0.78
1F	0.36	0.17	0.35	0.13	1.5	2.0	1.4
Mean	0.27	0.12	0.21	0.086	0.96	1.3	0.97
Std Dev	0.054	0.031	0.077	0.028	0.31	0.41	0.25
%RSD	20	26	37	33	32	32	26
SEMean	0.024	0.014	0.034	0.013	0.14	0.18	0.11
Min/Max	0.23 / 0.36	0.098 / 0.17	0.17 / 0.35	0.06 / 0.13	0.77 / 1.5	1.0 / 2.0	0.78 / 1.4

Appendix III

Results of spike recovery efficiency

To determine spike recovery efficiency, 0.5 mls of a standard mix (16 µg/ml) of DDT, DDD, DDE and dieldrin was added to fish sample #1, before the extraction procedure. Results are in mg/kg.

	DDT	DDD	DDE	Dieldrin
Unspiked Sample	0.21	0.09	0.96	ND
Spiked Sample	1.90	1.60	2.90	1.90
% Recovery	105.60	94.60	121.30	118.80

Appendix IV

Comparison of GC/EC and GC/MS results

These results are a comparison of average concentrations and ranges of mirex, photomirex, DDE and total DDT in fish tissue from analyses on both the GC/EC and GC/MS. DDT and DDD are not included, they are unstable and break down quickly; therefore any differences in GC/EC and GC/MS data observed could be erroneous. Numbers in parentheses are standard errors. Results are in mg/kg.

	Mirex	Photomirex	DDE	Total DDT
GC/EC	0.24 (0.038)	0.10 (0.016)	0.82 (0.29)	1.1 (0.17)
Range	0.095-0.48	0.040-0.20	0.32-1.7	0.43-2.2
GC/MS	0.24 (0.038)	0.11 (0.014)	0.79 (0.13)	0.99 (0.15)
Range	0.099-0.46	0.061-0.21	0.21-1.4	0.32-1.72

Appendix V

Field and analytical data for 1992 fall collections of Lake Ontario Chinook Salmon
Results are in mg/kg.

Sample #	Sex	Age (yr)	Weight (kg)	Length (cm)	Lipids (%)	Mirex	Photo-mirex	DDT	DDD	DDE	Total DDT	PCB Aroclor	Dieldrin
1	M	3	9.1	101.0	3.0	0.27	0.12	0.21	0.087	0.96	1.3	0.97	ND
2	M	2	7.9	87.0	2.8	0.18	0.065	0.11	0.033	0.52	0.66	0.53	ND
3	M	3	12.3	104.0	7.9	0.28	0.11	0.21	0.072	0.82	1.1	0.90	ND
4	M	3	8.6	95.0	2.4	0.34	0.15	0.25	0.11	1.3	1.7	1.3	ND
5	F	3	10.0	94.0	5.0	0.48	0.20	0.28	0.11	1.4	1.8	1.5	ND
6	F	3	7.5	86.0	1.9	0.13	0.051	0.087	0.027	0.40	0.51	0.50	ND
7	M	1	2.6	57.2	5.5	0.15	0.087	0.14	0.052	0.59	0.78	0.73	ND
8	M	3	10.3	95.5	2.2	0.25	0.11	0.18	0.083	0.98	1.2	1.0	ND
9	M	3	10.8	94.0	6.3	0.45	0.18	0.30	0.17	1.7	2.2	1.7	ND
10	M	1	2.0	60.5	4.0	0.091	0.040	0.10	0.030	0.33	0.46	0.33	ND
11	M	1	2.4	60.5	1.9	0.12	0.058	0.13	0.049	0.47	0.65	0.47	ND
12	M	1	1.9	56.0	3.7	0.095	0.040	0.079	0.028	0.32	0.43	0.32	ND
5 Eggs	--	--	--	--	12.7	0.22	0.11	0.21	0.085	1.4	1.7	1.2	0.059
6 Eggs	--	--	--	--	10.7	0.16	0.091	0.18	0.099	1.0	1.3	0.98	0.062

ND - Not Detected

Appendix VI

Data for 1977 Collection of Lake Ontario Coho and Chinook Salmon

Sample No.	Species	Sex	Age (yr)	Weight (kg)	Length (cm)	Lipid (%)	Mirex (mg/kg)
T4	Chinook	Male	NA	0.41	36.5	NA	0.16
T12	Chinook	Male	NA	1.25	48.0	NA	0.19
T15	Chinook	Male	NA	0.45	35.0	NA	0.19
K1	Coho	Male	NA	1.78	55.0	NA	0.090
K24	Coho	Male	NA	1.09	46.0	NA	0.10
K25	Coho	Male	NA	1.67	53.2	NA	0.070
T10	Chinook	Female	NA	1.039	46.5	NA	0.19
T11	Chinook	Female	NA	1.30	47.5	NA	0.28
K30	Coho	Male	NA	1.49	51.8	NA	0.20
K27	Coho	Female	NA	1.24	49.0	NA	0.13
K28	Coho	Female	NA	1.34	50.4	NA	0.16
K29	Coho	Female	NA	1.37	50.0	NA	0.17
T2	Chinook	Male	NA	2.81	65.0	NA	0.28
T1	Chinook	Male	NA	6.31	83.0	NA	0.34
T3	Chinnok	Male	NA	7.39	88.5	NA	0.41
K20	Coho	Male	NA	4.77	81.2	NA	0.22
K15	Coho	Male	NA	3.29	72.5	NA	0.23
K22	Coho	Male	NA	5.40	75.7	NA	0.27
T5	Chinook	Female	NA	5.92	83.0	NA	0.30
T8	Chinook	Female	NA	4.30	63.9	NA	0.23
T9	Chinook	Female	NA	7.75	89.7	NA	0.34
K13	Coho	Female	NA	5.36	76.3	NA	0.29
K14	Coho	Female	NA	4.07	72.2	NA	0.31
K19	Coho	Female	NA	5.087	72.6	NA	0.19

NA - Not Available

Appendix VII

Data for 1982 Collection of Lake Ontario Coho and Chinook Salmon

Sample No.	Species	Sex	Age (yr)	Weight (kg)	Length (cm)	Lipid (%)	Mirex(mg/kg)
T1	Chinook	Female	NA	2.35	52.4	NA	0.23
T2	Chinook	Female	NA	3.59	68.0	NA	0.14
T3	Chinook	Female	NA	1.45	56.8	NA	0.079
T4	Chinook	Male	NA	1.59	55.3	NA	0.046
T5	Chinook	NA	NA	0.91	45.1	NA	0.031
T6	Chinook	Female	NA	6.72	80.6	NA	0.27
T8	Chinook	Male	NA	5.54	79.3	NA	0.32
T9	Chinook	Male	NA	1.53	51.0	NA	0.15
T10	Chinook	Female	NA	2.92	61.1	NA	0.22
T11	Chinook	Male	NA	2.71	65.5	NA	0.102
T12	Chinook	Female	NA	10.58	89.5	NA	0.32
T13	Chinook	Female	NA	7.43	83.9	NA	0.35
K1	Coho	Male	NA	3.81	67.3	NA	0.102
K2	Coho	Female	NA	5.81	79.3	NA	0.15
K3	Coho	Female	NA	5.67	77.9	NA	0.19
K4	Coho	Male	NA	5.09	72.4	NA	0.27
K5	Coho	Male	NA	1.68	53.8	NA	0.22
K6	Coho	Male	NA	4.21	70.1	NA	0.19
K7	Coho	Male	NA	0.46	35.2	NA	0.102
K13	Coho	Male	NA	0.63	37.2	NA	0.091
K12	Coho	Male	NA	0.70	39.9	NA	0.099
K14	Coho	Male	NA	5.61	77.1	NA	0.21
K16	Coho	Male	NA	3.41	64.1	NA	0.19
K18	Coho	Female	NA	4.11	76.9	NA	0.26

NA - Not Available

Appendix VIII

Data for 1986 Collection of Lake Ontario Coho and Chinook Salmon

Sample No.	Species	Sex	Age (yr)	Weight (kg)	Length (cm)	Lipid (%)	Mirex (mg/kg)
K1	coho	M	1+	0.34	37.3	6.53	0.12
K2	coho	M	2+	2.27	62.2	3.22	0.13
K3	coho	M	2+	3.40	71.5	3.86	0.12
K4	coho	M	2+	2.84	66.7	3.16	0.20
K5	coho	F	2+	3.18	68.5	6.28	0.32
T6	chinook	M	3+	8.40	90.2	0.35	0.13
T7	chinook	M	1+	1.82	52.8	6.10	0.067
T8	chinook	M	1+	1.82	53.0	3.41	0.12
T9	chinook	M	1+	2.04	55.4	5.84	0.10
T10	chinook	M	3+	9.08	97.2	1.09	0.31
T11	chinook	M	3+	9.53	90.2	2.11	0.41
T12	chinook	F	3+	9.76	94.0	1.95	0.40
T13	chinook	F	3+	11.12	100.3	2.27	0.34
T14	chinook	F	2+	6.70	81.3	1.75	0.18
T15	chinook	M	2+	5.22	72.5	4.79	0.18
T16	chinook	M	2+	6.81	82.6	9.05	0.22
T17	chinook	M	1+	2.04	54.0	3.46	0.20
K18	coho	M	3+	4.54	78.7	2.12	0.20
K19	coho	M	3+	5.33	80.0	2.96	0.23
K20	coho	M	3+	4.09	74.9	2.85	0.22
K21	coho	M	3+	3.86	75.7	2.88	0.20
K22	coho	F	1+	1.70	59.7	1.35	0.12
K23	coho	M	1+	0.68	45.0	1.69	0.032
K24	coho	M	1+	0.34	39.4	1.56	0.015